Analysis of the Purity and Storage Stability of L-Ascorbic Acid Powders

Application Note

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Introduction

Most previous studies of the storage stability of L-ascorbic acid were done on solutions of the acid. However, L-ascorbic acid is usually kept in the dry powder form, batches of which may contain breakdown products or other impurities arising from the production process. The presence of these contaminants is suspected to adversely affect the stability of the acid and/or contribute to color changes in the stored product over time.

This note describes a trial of the purity and stability of stored L-ascorbic acid powder samples from four different manufacturers, and assesses degradation compounds and/or precursors from the production process. Ion suppression reversed phase HPLC was used, with PLRP-S columns. PLRP-S is a rigid macroporous styrene/divinylbenzene HPLC phase with outstanding chemical and physical stability with acidic eluents.

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Materials and Reagents

Samples: L-ascorbic acid powders from different manufacturers produced from 1971 to 1987
Impurity reference compounds: L-dehydroascorbic acid (DHAA), 2,3-diketo-L-gulonic acid, L-threonic acid, oxalic acid, furfural, 5,6-*O*-isopropylidene-L-ascorbic acid, D-gulonic acid-1,4-lactone

Conditions

Column: PLRP-S 100Å 5 µm, 150 x 4.6 mm (p/n PL1111-3500)
Eluent: 0.2 M NaH_2PO_4, pH 2.14
Flow rate: 0.5 mL/min
Detector: UV, 268 and 220 nm

Results and Discussion

A plot of manufacturing date against purity is given in Figure 1, showing a gradual decline in L-ascorbic acid with age even in dry powder formulations. Comparison of the retention times of sample peaks with those of the reference compounds (Table 1) did not reveal any sign of degradation products or precursors associated with the manufacture or storage of L-ascorbic acid. The absence of these compounds suggests that other degradation routes are present when L-ascorbic acid is stored under near anaerobic conditions of low water activity.

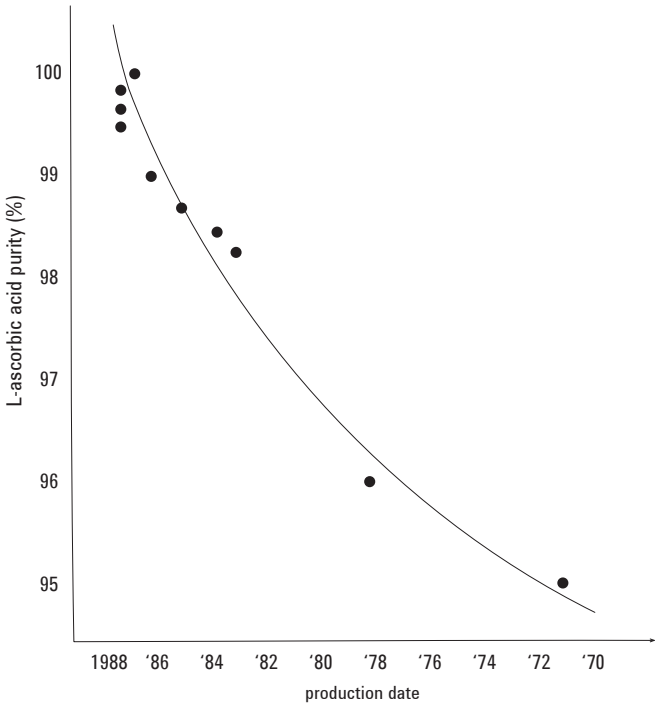

Figure 1. Graph of L-ascorbic acid purity and production date of powder samples of L-ascorbic acid.

Table 1. Retention times of reference compounds.

Sample	L-ascorbic acid association	Retention time (min)	Limit of detection (µmol)
oxalic acid	degradation product	6.00	0.039
L-threonine	degradation product	6.30	0.053
2,3-diketo-L-gulonic acid	precursor	6.60	-
D-gulonic acid-1,4-lactone	precursor	6.60	-
L-dehydroascorbic acid	degradation product	7.40	0.017
L-ascorbic acid	-	8.70	
5,6-*O*-isopropylidene-L-ascorbic acid	precursor	8.70	
fufural	degradation product	12.10	

The samples varied in their degree of whiteness regardless of the date of production, suggesting that differences in whiteness were not indicators of purity but may have been be due to differing production methods. However, the aged samples manufactured in 1971 and 1978 had a brown appearance, most evident in the 1978 sample. Measurement of the L-ascorbic acid content of these samples was lower compared to newer samples, but no impurities were detected.

The oxidative degradation of a solution of L-ascorbic acid was assessed, following the pathway shown in Figure 2.

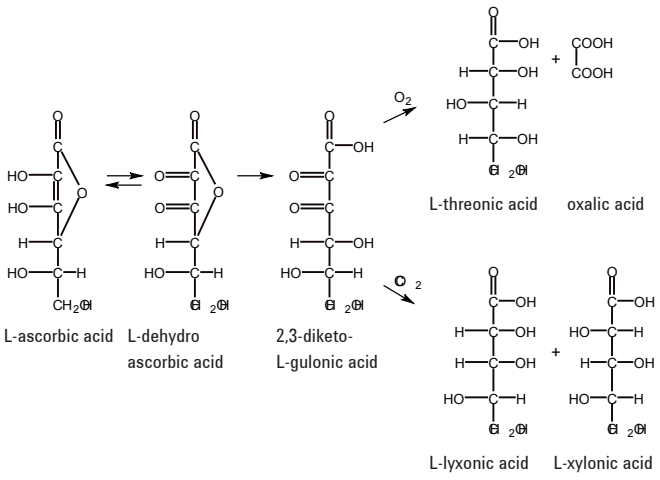

Figure 2. The proposed oxidative degradation path of L-ascorbic acid.

Table 1 shows the minimum limits of detection of the degradation products L-dehydroascorbic acid, oxalic acid and L-threonic acid from this assay. It was not possible to carry out a test for 2,3-diketo-L-gulonic acid because a pure reference compound was not available.

The complete data set and analysis is available in Kennedy *et al.* (1989).

Conclusion

Ion suppression HPLC using PLRP-S columns successfully elucidated the effects of age on the gradual deterioration of dry powder L-ascorbic acid, and suggested the presence of alternative degradation mechanisms when the acid is stored in dry powder form.

Reference

Kennedy, JF, Rivera, ZS, Lloyd, LL, Warner, FP, White, CA (1989) The use of reversed-phase HPLC for the determination of purity and stability on storage of L-ascorbic acid samples. *J. Micronutr. Anal.,* 5, 281-289.

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